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THE EFFECT OF 300°C STEAM ON LUBRICATED AND PRELOADED ASTM A-193 B7 BOLTING

Report No. 91-39-K

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ABSTRACT

Ten candidate thread lubricants for use in nuclear service were screened for Stress Corrosion Cracking (SCC) effects in tensioned ASTM A-193 B7 bolting. Forty-four fastener specimens were tensioned to 50% and 90% of yield strength to simulate moderate and high stress conditions, and aged in a 300°C steam autoclave environment. Neither lubricated bolts nor as-received bolting showed SCC damage, however, based on relative performance, "Thread Guard" and "Fel-Pro" premium grade copper and nickel based thread lubricants were rated as preferred products.

Based on torque/turn measurements, turn angle shows promise as an estimator of bolt preload and further in-plant torque/turn testing is recommended. A reconsideration of B7M as a replacement for B7 and more aggressive accelerated aging testing to produce SCC are also recommended.

sub	file	date	report no.
32-050-020	831.1-7	March 19, 1991	91-39-K

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EXECUTIVE SUMMARY

THE EFFECT OF 300°C STEAM ON LUBRICATED AND PRELOADED ASTM A-193 B7 BOLTING

W.J. Janis

Thread lubricants have been implicated in Stress Corrosion Cracking (SCC) failures of bolting materials. Of particular concern to Bruce NGS-B personnel is the large number of thread lubricants used at the station and the potential of some of these materials to promote SCC in steam or high temperature water pipe flange fasteners, where ASTM A-193 B7 bolting may be exposed to a steam environment and may also be under high preload tension.

Ten candidate thread lubricants for use in nuclear service were screened for SCC-promoting effects in tensioned ASTM A-193 B7 bolting. Fastener specimens were tensioned to 50% and 90% of yield strength to simulate moderate and high stress conditions and all fastener assemblies were aged at one time in a 300°C steam autoclave environment.

Stress Corrosion Cracking was not induced in any of the test specimens, including as-received bolting with no thread lubricant, and therefore no lubricant was revealed as an aggressive SCC accelerator under the test conditions.

A relative lubricant effectiveness rating was possible based on secondary effects such as assembly torque, post-test unlocking torque and post-test bare metal contact area in the load zone.

The secondary effects data indicate that premium grade copper based lubricants outperform nickel based products. Copper and nickel based lubricants outperform aluminum, iron and molybdenum based materials.

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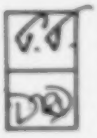
Based on relative performance, "Thread Guard Nickel", "Fel-Pro N-5000", "Thread Guard Copper" and "Fel-Pro N-1000" are considered the only qualified products for applications requiring copper and nickel based thread lubricants.

Torque and turn angle data show that turn angle is a better measure of bolt tension than torque, and in-plant torque/turn experiments with real or mock-up joints, complete with gasketing and washers, are therefore recommended.

Further aging studies are also recommended to develop an accelerated aging method which can produce SCC damage in lubricated fastener specimens in 1,000-2,000 hours.

Based on published SCC data, it is further recommended that ASTM B-7M material be reconsidered as a replacement for B7, if other considerations such as reduced yield strength and possible misapplication can be adequately addressed.



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To: Mr. R.W. Pockett
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THE EFFECT OF 300°C STEAM ON LUBRICATED AND PRELOADED ASTM A-193 B7 BOLTING

1.0 INTRODUCTION

Stress corrosion cracking (SCC) of bolting materials used in Ontario Hydro is a well documented occurrence. For example, Metallurgical Research Reports M89-34-K, M85-77-K and M84-170-K detail Motorized Valve Bonnet Stud failures at BNGS-B, and HP Turbine Stub Shaft Bolting failures at PNGS-A, respectively.

For SCC to take place, there are three prerequisites: (1) the material must be a type that is sensitive to SCC; (2) the load stress must exceed a certain minimum value; and (3) the environment must be conducive to SCC.

It is known that A-193 B7 material is metallurgically sensitive to SCC, and Ontario Hydro station operating experience suggests that not all overloading can be prevented under field conditions. Therefore, for some bolts, two of the three necessary conditions for SCC are satisfied at the time of fastener assembly. If the local environment is also sufficiently aggressive, A-193 B7 fasteners may be vulnerable to SCC failure.

Of particular concern to BNGS-B personnel is possible stress corrosion cracking of lubricated, ASTM A-193 B7 bolting used on flanges where leakage has exposed the fasteners to a wet steam environment. The weakening of such piping and valve joints could seriously compromise system integrity and increase the likelihood of personal injury in the event of joint failure.

Literature reports have implicated molybdenum disulphide and tramp elements such as lead, sulphur, fluorine and chlorine in anti-seize lubricants as SCC accelerators/1-4/. Several "moly" products and a number of other thread lubricants based on copper, nickel, and graphite blends are in use at Ontario Hydro

generating stations and it is possible that some of these products accelerate SCC in A-193 B7 bolting under high moisture and load conditions.

A meeting of personnel from BNGS-A, BNGS-B, Mechanical Design, Supply Division and Research Division was held in September, 1989, to address the above concerns/5/. It was concluded that the number of thread lubricants used at Ontario Hydro stations should be reduced to a minimum and that the products used in future should be screened for SCC-promoting effects.

Subsequently, Mechanical Design reviewed the BNGS-B thread lubricant inventory and produced a consolidated list of the most promising candidate lubricants for bolting applications/6/. Under the direction of BNGS-B, and with input from Mechanical Design and Metallurgical Research, a testing program for the candidate lubricants on ASTM A-193 B7 bolting was developed by the Materials Chemistry Section/7/.

This report describes the testing program and discusses the results.

2.0 TEST METHODS AND MATERIALS

2.1 Method Development and Test Conditions

Accelerated aging methods vary for the study of SCC in bolting. However, it is clear from the literature that high temperature, high loading and excess moisture are required if failures are to be seen within a few months.

Previous SCC bolting studies have used autoclaves to age bolted test assemblies or notched bolting specimens in water or steam environments. Temperatures ranged from 80°C to above 300°C, durations were from 1000 to 5000+ hours and specimens were strained either statically in preloaded assemblies or dynamically during an aging test at a constant strain rate/1,4/. Environmental conditions such as lubricant type, water pH and tramp trace element content were varied as required.

Based on the above, the general test plan adopted for this work was to age mechanically strained, bolting assemblies under "worst case" high temperature and humidity conditions in an autoclave and examine the specimens for SCC damage after a 1000 hour aging period.

Based on the literature reports, an autoclave test temperature of 300°C was selected for this project. This temperature was considered high enough that evidence of SCC would be detectable in sensitive systems in about 1000 hours, and it was within the pressure capabilities of standard autoclaves available at Ontario Hydro Research Division (OHRD).

of the large number of lubricants to be tested,
essed assemblies were considered more appropriate for this
than dynamically loaded assemblies. Dynamical systems
ly handle only one bolting specimen at a time per
ve and are not available at OHRD.

r feed water steam environment was selected as the
ving environment to mimic station conditions at a steam
Boiler feed makeup water was provided by Bruce NGS-B for
rpose.

ely small, 7.6 cm x 1.3 cm dia. (3" x 0.5" dia.), bolting
ns were selected for testing as these were considered
nough to be representative of flange bolting and yet small
to allow all the required lubricant/preload combinations
valuated in one test using a single 4 L autoclave.

late field assembly conditions, fastener components were
aned prior to application of a lubricant. That is, the
eatment thermal black present on "as-received" ASTM A-193
s was not removed.

stener Specimens and Assembly Procedure

st fastener consisted of the following:

7.6 cm x 1.3 cm (3" x 1/2") ASTM A 193 Grade B7
-thread stud.

1.3 cm (1/2") ASTM A 194 Grade 2H Hvy Hex. Nuts.

2.7 cm o.d. (1 1/16") x 0.24 cm (3/32") carbon steel
ers.

3.8 cm (1 1/2") x 2.5 cm (1" o.d.) Sch. 80 pipe spacer,
cm i.d. (1/2").

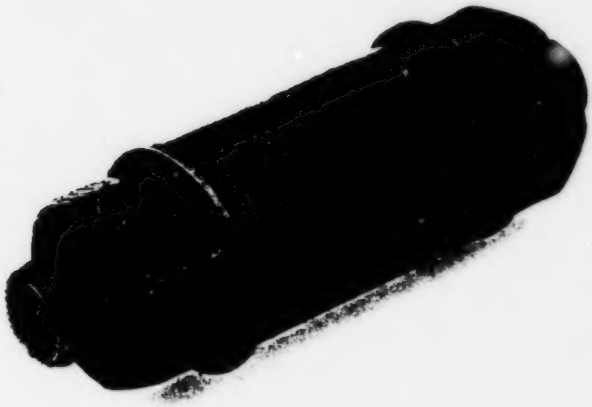
s were loaded against the washers which were loaded
the pipe spacer in the middle of the assembly. The bolt
resulting fastener was therefore under tensile and
al stress, while the nuts, washers and spacer were under
sion. Photographs of bolting, fastener components and an
ed fastener are shown in Figure 1.

semblies were prepared for each lubricant, two tensioned
of the bolt yield strength (YS) and two to 50% of yield
n. Ninety percent YS represents an over-tensioned
on while fifty percent YS represents the acceptable
r condition.

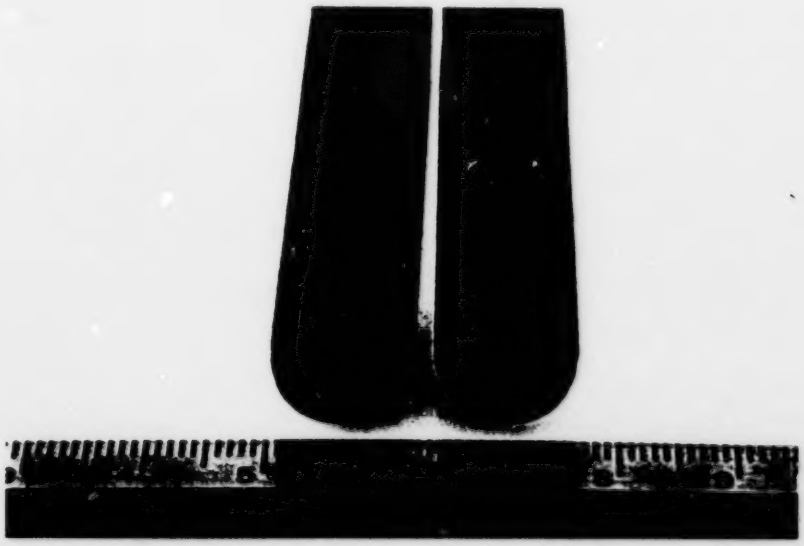
FIGURE 1

Bolting and Test Specimens

(A) Assembled Test Fastener



(B) Bolting - End Ground Flat and As Received
(Scale is in cm)



The tension values are based on those used in previous studies, which have used stresses in the 80-95% of YS range to simulate an over-stressed condition.

A benchmark bolt yield stress for the ASTM A-193 B7 bolting specimens was determined experimentally from tensile strength tests on "as received" bolting. As described in Appendix 1, the resulting YS value and Young's modulus were then used to calculate the bolt elongation required to achieve the selected tensions.

Based on the Appendix 1 calculations, the extensions required for 50% and 90% YS were approximately 5 and 9 thou. (0.127 mm and 0.229 mm), respectively, and these two extensions were adopted as standard for this study.

Bolt lengths and extensions were measured with a micrometer, and the ends of all bolts were ground flat to improve the accuracy of the elongation measurements. Prior to assembly, the lubricants were brushed onto both ends of the bolts, including the area under the final resting positions of the nuts in the assembled test fasteners.

The torque and angle required to achieve a given extension were measured for all specimens at the time of fastener assembly. Torques were measured with a dial gauge torque wrench. Angles were measured relative to a protractor fixed to one nut, using a pointer fixed to the torque wrench applying force to the other nut. Nuts were snugged tight by hand before wrench torque was applied.

2.3 Test Lubricants

The lubricants chosen for testing are presented in Table I, below. These include those proposed by the Materials & Welding Unit (Reference 6) plus a moly-type reference and "as received" and "specially cleaned" blanks. The nickel based materials are for use at high temperature (up to about 1450^{o}C) and severe conditions. Product literature claims that these products are compatible with all metals likely to be encountered in nuclear plants.

The copper based materials are for use at temperatures up to about 600^{o}C and are acceptable for high nickel alloy materials.

The Hi-Temperature products are of lesser quality than the nuclear grade materials and may contain tramp elements. In addition, trace element analysis of the Hi-Temperature Thread Guard and Chesterton products shows them to be based on Aluminum and Iron, respectively, rather than Nickel and Copper.

The G-n paste is a standard MoS_2 compound to be used as a benchmark material and the "as received" bolt is unlubricated, but coated with a thermal black oxide as a result of heat treating.

Table I

SCC Test Program Test Lubricants and Blanks

Sample Number	Name	Anti-seize Description	Abbreviation
1	Thred Guard Nickel	Premium Nickel	TG-Ni
2	Fel-Pro N-5000	Premium Nickel	FP-Ni
3	Chesterton #772	Premium Nickel	CH-Ni
4	Thred Guard Copper	Premium Copper	TG-Cu
5	Fel-Pro N-1000	Premium copper	FP-Cu
6	Polybutylcuprisil	Premium Copper	PBC
7	Thred Guard GP	General Hi-Temp Iron	TG-Fe-GP
8	Fel-Pro C5-A	General Hi-Temp Copper	FP-Cu-GP
9	Chesterton #785	General Hi-Temp Aluminum	CH-Al-GP
10	G-n Paste	Molybdenum disulphide	MOLY
11	Blank	Bolt as rec'd (thermal black coat)	BLANK
12	Blank - Cleaned	Solvent Cleaned Blank	BLANK (cleaned)

Table II presents approximate total solids and major/minor trace element data for the test lubricants. The total solids were determined by weighing the lubricant samples before and after two extractions with naphtha and therefore may contain some non-naphtha-soluble organics as well as the inorganic solids. The elemental analyses were performed by Atomic Emission Spectrometry (AES) and X-ray Fluorescence (XRF).

Major elements are present in percent quantities, minor elements are estimated to be present in quantities < 1000 mg/kg (parts per million).

Two methods for elemental analysis were used for greater assurance, because both methods give only approximate quantitative values for trace elements. AES suffers from the paste-like nature of the products and both AES and XRF suffer from interferences between the major constituent metals and the trace materials.

The following are noted from the analytical data:

(1) All materials except FP-Ni contain trace contaminants.

(2) PBC contains lead in high concentration.

(3) Trace sulphur is present in samples 4,6,7 and 8.

(4) Graphite is present in most, if not all, candidate lubricants.

(5) Halogens were not detected.

Table II

SCC Test Program Test Lubricants

Sample Number	Name	Solids (%)	Major/Minor Elements
1	Thred Guard Nickel[1]	50	Ni/Si,Al,Fe
2	Fel-Pro N-5000[1]	55	Ni/-
3	Chesterton #772	60	Ni/Si,Al,Fe
4	Thred Guard Copper	60	Cu/S,Si
5	Fel-Pro N-1000[1]	55	Cu/Si,Al
6	Polybutylcuprisil	15	Cu,Pb/S,Si,Al,Fe
7	Thred Guard GP[1]	20	Fe,Ca/Si,S,Al
8	Fel-Pro C5-A[1]	25	Cu/S,Ca,Zn
9	Chesterton #785	65	Al/Si,Fe,Ca
10	G-n Paste[1]	60	Mo,S/Zn,Ca,Si

[1] *Product literature indicates that graphite is present.*

2.4 Post-Aging Analysis

After autoclaving for 1000 hours at 300°C, the fastener specimens were disassembled using a torque wrench to estimate maximum breakaway torque.

The bolts were cleaned ultrasonically, and the previously lubricated areas were inspected for gross cracks and pits using low and high power microscopes.

Yield strengths were then determined using a tensile strength machine.

3.0 RESULTS

3.1 Preloading Torque and Turn Angle

The measured torques and turning angles required to achieve 50% YS and 90% YS tensile preload during assembly of lubricated and unlubricated fasteners are presented in Appendix 2 and discussed below.

3.1.1 Preloading Torque Versus Bolt Tension

The preload torque data for the lubricated assemblies are summarized in Figures 2 and 3 for nickel, molybdenum and aluminum based lubricants, and copper and iron based thread lubricants, respectively. Linearity of these two point curves over the 50-90% YS range is assumed, based on stiffness considerations (see Sections 3.1.2 and 3.1.3).

Data for unlubricated and lubricated blanks are also presented on Figure 2, and points representing recommended torques for lubricated and unlubricated A-193 B7 material are marked on both Figures (torque values from Reference 8).

The following are noted:

(1) Except for PBC and TG-Fe-GP, the slopes of the lubricated fastener curves are similar to that of the as-received "blank" fastener curve. The average slope is approximately 1.0 N·m (0.75 Ft-Lbs) torque/percent preload tension, measured as a percent of yield.

(2) The curve for the as-received blank approximates the average of the lubricated bolting curves, indicating no major difference in friction between the residual thermal black coating of the blank and that of the average thread lubricant.

(3) The solvent-cleaned blank shows the highest required torque at both 90% and 50% YS for all but PBC at 50% YS. This is as expected for the solvent cleaned and wiped surface, discounting the PCB anomaly, as the clean surface should have a minimal carbonaceous heat treating residual (lubricating film) and maximum base metal oxide (high friction) surface.

(4) The points representing recommend torques for unlubricated B7 bolting material at 80% YS encompass most of the curves for lubricated fasteners, and also the curve for the as-received blank. The experimental points are therefore consistent with standard torquing guide predictions for most products.

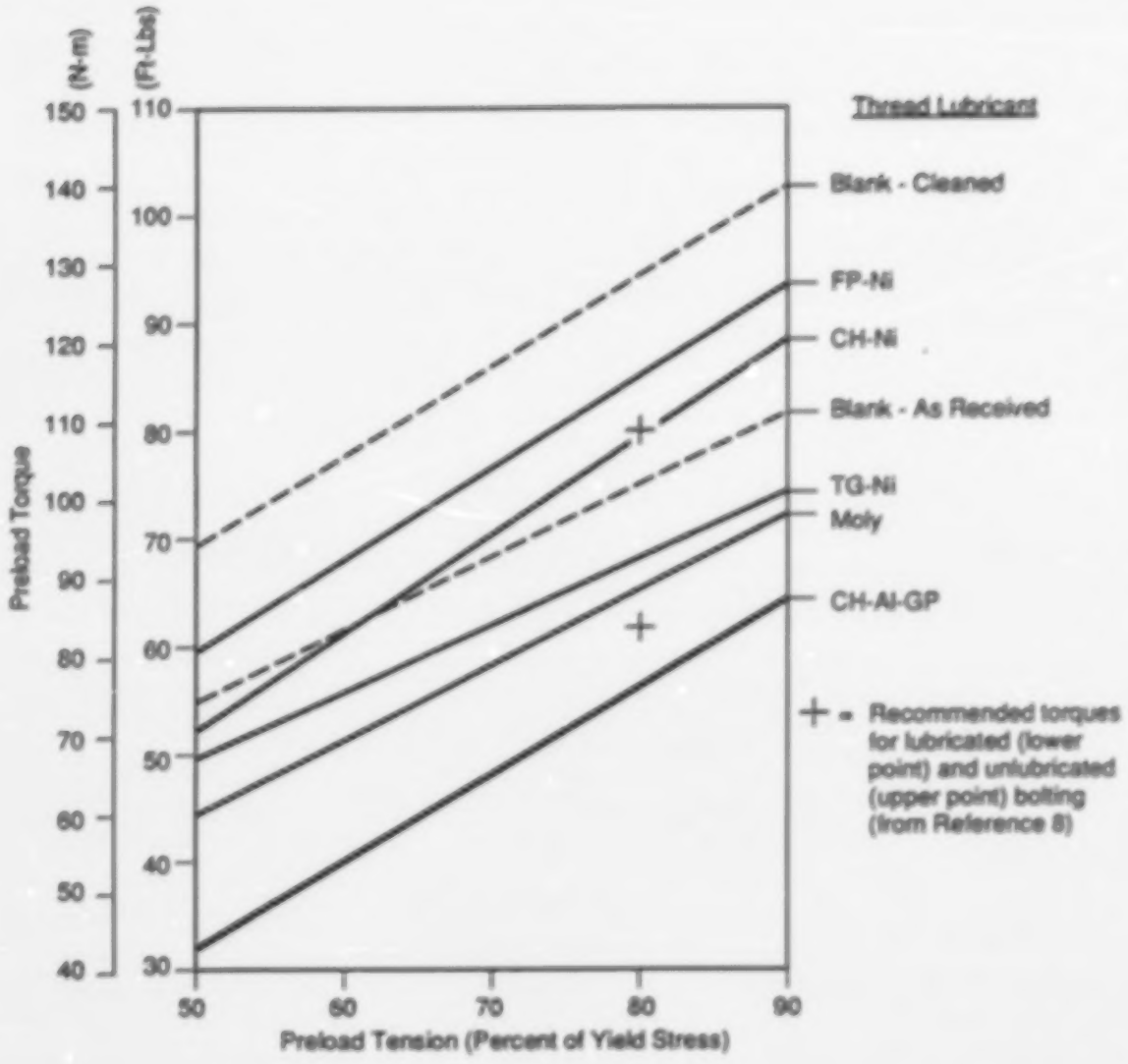


FIGURE 2

PRELOAD TORQUE VERSUS PRELOAD TENSION - NICKEL, MOLYBDENUM AND ALUMINUM THREAD LUBRICANTS ON ASTM A-193 B7 BOLTING

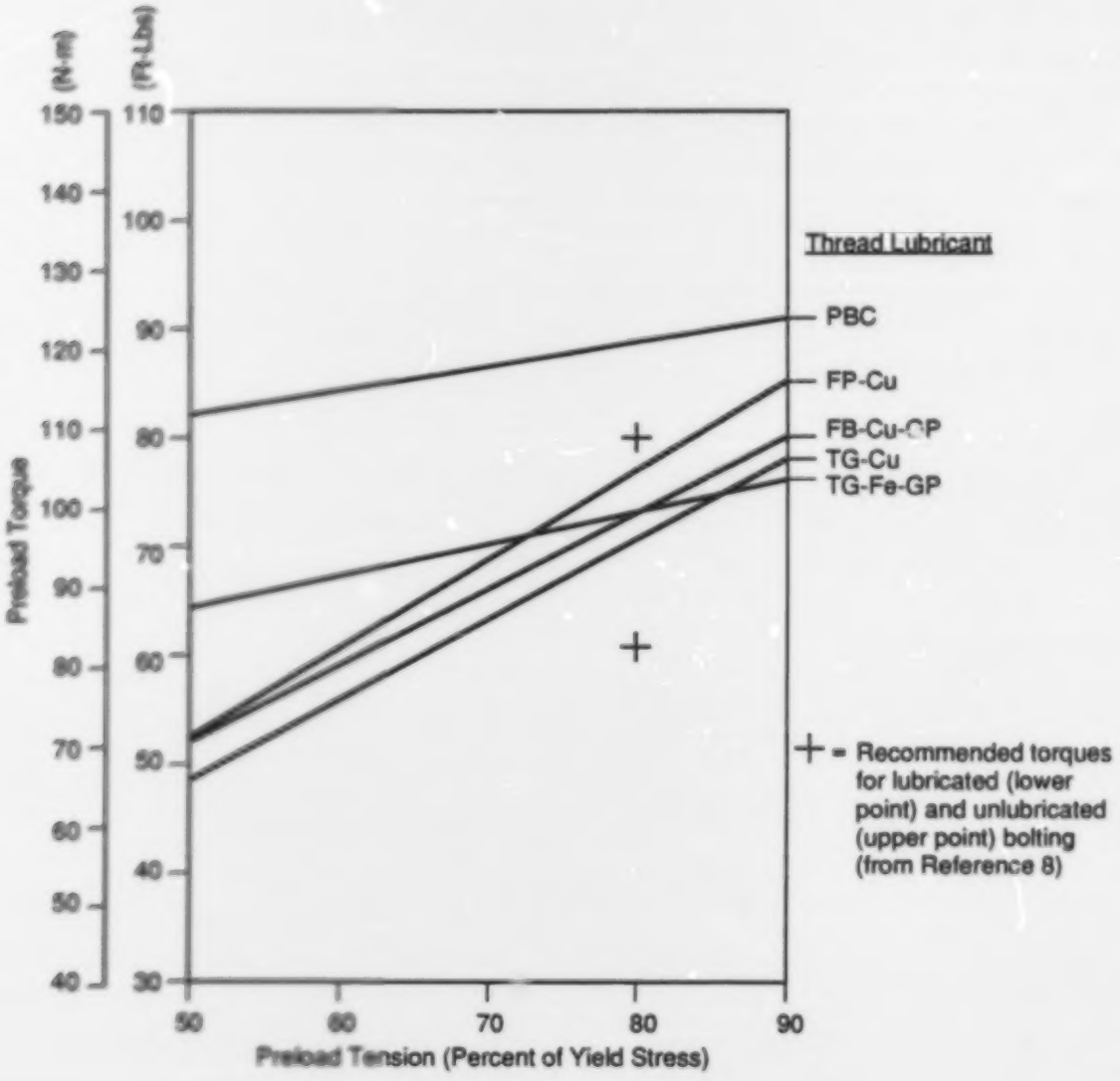


FIGURE 3

PRELOAD TORQUE VERSUS PRELOAD TENSION - COPPER AND IRON THREAD LUBRICANTS ON ASTM A-193 B7 BOLTING

(5) Preload torque variability at 50 and 90 percent of yield stress is approximately 40% and 20% of the respective average values. This indicates that torque improves as an indicator of bolt tension as the stress increases. However, as expected, torque is not a particularly good indicator of bolt tension.

Table III lists significant bolt tension ranges for fasteners, as identified in References 8 and 9. These references, and Reference 10, note that industrial practice favors 60-70 percent of Tensile Strength (TS) or, as shown in Appendix 1, 70-80 percent of the yield strength (YS), as an optimum tensioning range.

Table III

Bolt Tension Ranges of Significance

Tension (Percent of YS)[1]	Range Description
55-70	Ideal Tension - includes a safety factor
48-76	Acceptable Tension - no safety factor
34-93	Widest possible range - some torsion loss possible at high end.
< 34	Unstable - can vibrate loose
> 93	Unstable - SCC, yield in tension, rupture

[1] YS = Yield Strength. Reference 8 data is expressed in terms of Tensile Strength. See Appendix 1 for the conversion to percent of Yield Strength.

Based on the above, most of the tension range spanned by the experimental tests is considered acceptable. Specifically, the range: 50-80 is viewed as acceptable while the range: 81-90 is viewed as doubtful.

From Figures 2 and 3, the guide recommended torque is 84.3 N·m (62 Ft-Lbs). Therefore all lubricants which give tensions between 50 and 80% of yield at 84.3 N·m (62 Ft-Lbs) are acceptable, with those lubricants having tensions between 70 and 80 percent of yield being preferred.

The test lubricants can therefore be rated as shown in Table IV, assuming that the lubricants which most closely match the torque table guide are the most acceptable. Note that the non-standard Al, Fe and Cu/Pb (PBC) based lubricants rank poorly relative to Ni, Cu and Mo based products. The ranking data are discussed more fully in combination with other ranking data in Section 4.1.

Table IV

Lubricant Rating Based on Pre-Load Considerations

Category	Lubricant	YS(%)	Rank (1=Best)
OPTIMUM (70-80% YS @62 Ft-Lbs)	MOLY	75	1
ACCEPTABLE (50-69% YS @62 Ft-Lbs)	TG-Ni	68	2
	TG-Cu	66	3
	FP-Cu-GP	62.5	4
	CH-Ni	60	5
	BLANK	60	5
	FP-Cu	60	5
	FP-Ni	50	6
PRELOAD TOO HIGH (>80% YS @62 Ft-Lbs)	Ch-Al-GP	86	7
PRE-LOAD TOO LOW (<50% YS @62 Ft-Lbs)	TG-FE-GP	<50	7
	PBC	<<50	8

The loading torque data of Appendix 2 were pooled according to metal type and analyzed statistically, as described in Appendix 3. Regardless of loading, preload torques were statistically indistinguishable for copper, molybdenum, and nickel based lubricants, and also non-lubricated assemblies. That is, no preload advantage is seen in using an anti-seize based on a specific metal type.

3.1.2 Preloading Turn Angle Versus Bolt Tension

A linear least squares fit of elongation to turn angle yields the following relation for the data of Appendix 2:

$$E = 2.6 + 0.127\ (\theta - 70) \qquad [1]$$

where E is the elongation (thou) and θ is the turn angle (degrees). In terms of bolt tension, this relation is:

$$T = 2.6 + 1.27\ (\theta - 70) \qquad [2]$$

where T is the tension as a percent of yield strength.

The correlation coefficient for the fit is 0.961, indicating good linearity. The standard deviation is approximately 10%, indicating less scatter than for torque versus tension curves.

The good fit between turn angle and bolt tension was expected, as the turn angle should be relatively independent of the lubrication used. In addition, the test assemblies used in this study are relatively stiff and resist compression, therefore most of the turning action results in bolt elongation.

A linear relation such as [2] cannot be assumed to apply to BNGS-B bolting however, as compression of gaskets and flange materials might be significant. Field or laboratory tests using actual flange materials are required to validate the use of angle measurement as a predictor of bolting preload.

3.1.3 Calculated Friction Coefficients

Average friction coefficients were calculated from the following equation relating applied torque to preload (From Reference 9):

$$T_{in} = F_p(P/2\pi + \mu_r r_t/\mathrm{Cos}(\theta) + \mu n r_n) \qquad [3]$$

where T_{in} = applied torque, F_p = preload, P = thread pitch, μ_r and μ_n = nut/bolt and nut/washer friction coefficients, r_t and r_n = thread/nut and nut/washer effective contact radii and θ = thread half-angle.

The calculated friction coefficients agree fairly well with the literature values and the calculated values would therefore have validity when used in torque calculations. However, as noted in Section 3.1.2, turn angle is probably a better predictor of bolt tension than torque for stiff systems.

Calculations are detailed in Appendix and the results are summarized in Table V.

3.2 Post-Aging Torque and Tensile Strength

After aging at 300°C for 1000 h in a boiler feed water environment, the fastener specimens were disassembled, cleaned and examined, and the tensile strengths of the bolts were determined.

3.2.1 Unlocking Torques

The measured torques required to unlock the bolting assemblies after aging are presented in Appendix 2 and discussed below.

The unlocking torque data for the lubricated assemblies are summarized in Figures 4 and 5 for nickel and molybdenum and

aluminum based thread lubricants, and copper and iron thread lubricants, respectively. Data for as-received blanks are also presented on Figure 4.

Table V

Calculated Friction Coefficients

Lubricant Lubricant	Friction (This Study)	Coefficient[1] (Literature Value)
CH-Al-GP	0.070	
BLANK	0.085	-
MOLY	0.093	0.098[2]
TG-Cu	0.100	-
TG-Ni	0.102	-
FP-Cu-GP	0.106	0.118[2], 0.10[3]
FP-Cu	0.113	0.116[2], 0.10[3]
CH-Ni	0.115	-
TG-Fe-GP	0.121	-
FP-Ni	0.129	0.10[3]
PBC	0.173	-
CLEAN BLANK	0.133	0.131[3]

1 Estimated accuracy is +/- 0.02.
2 AECL Data (Report SP-MISC 75) adapted from CRNL-903.
3 Supplier's data, or calculated from Supplier's data.

The following are noted:

(1) As expected, the unlocking torques are higher than the preloading torques. (Compare Figures 2 and 3 to 4 and 5, respectively).

(2) The MOLY lubricant behaves almost identically to the as-received blank bolting.

(3) On-average, unlocking torques for the copper based anti-seize compounds do not increase as much as those for nickel based compounds with increasing preload tension. Two of the copper based compounds, FP-CU and FP-Cu-GP, actually showed lower breakaway torques for the 90% YS specimens than for 50% YS specimens.

(4) More curve crossovers are apparent in Figures 4 and 5, compared with the curves of Figures 2 and 3. Some lubricants are obviously affected more than others by 300°C aging.

Assuming that low unlocking torque is desirable and that a preload stress of 75% YS is representative of bolting in the field, the lubricants can be rated relative to their expected

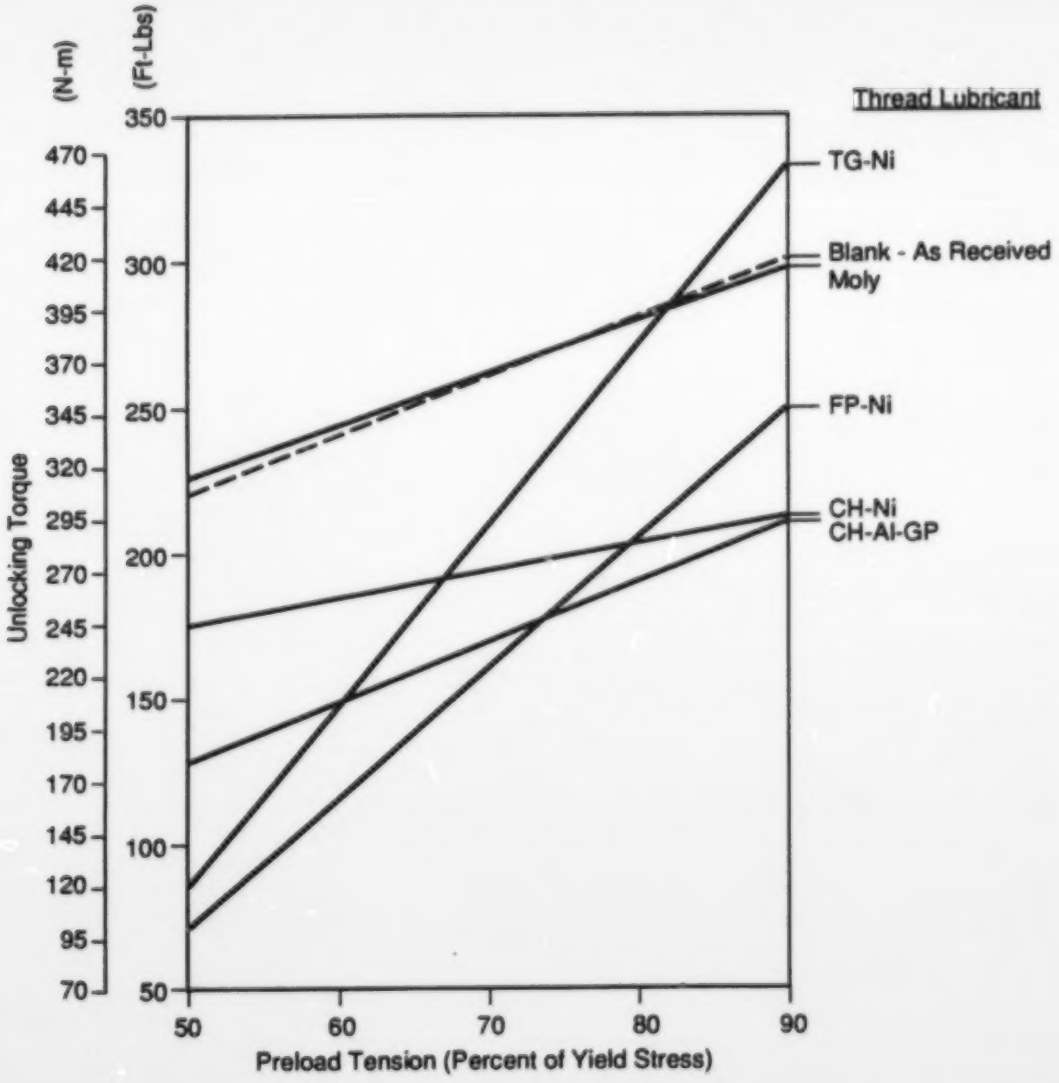


FIGURE 4

UNLOCKING TORQUE VERSUS PRELOAD TENSION - NICKEL, MOLYBDENUM AND ALUMINUM THREAD LUBRICANTS ON ASTM A-193 B7 BOLTING

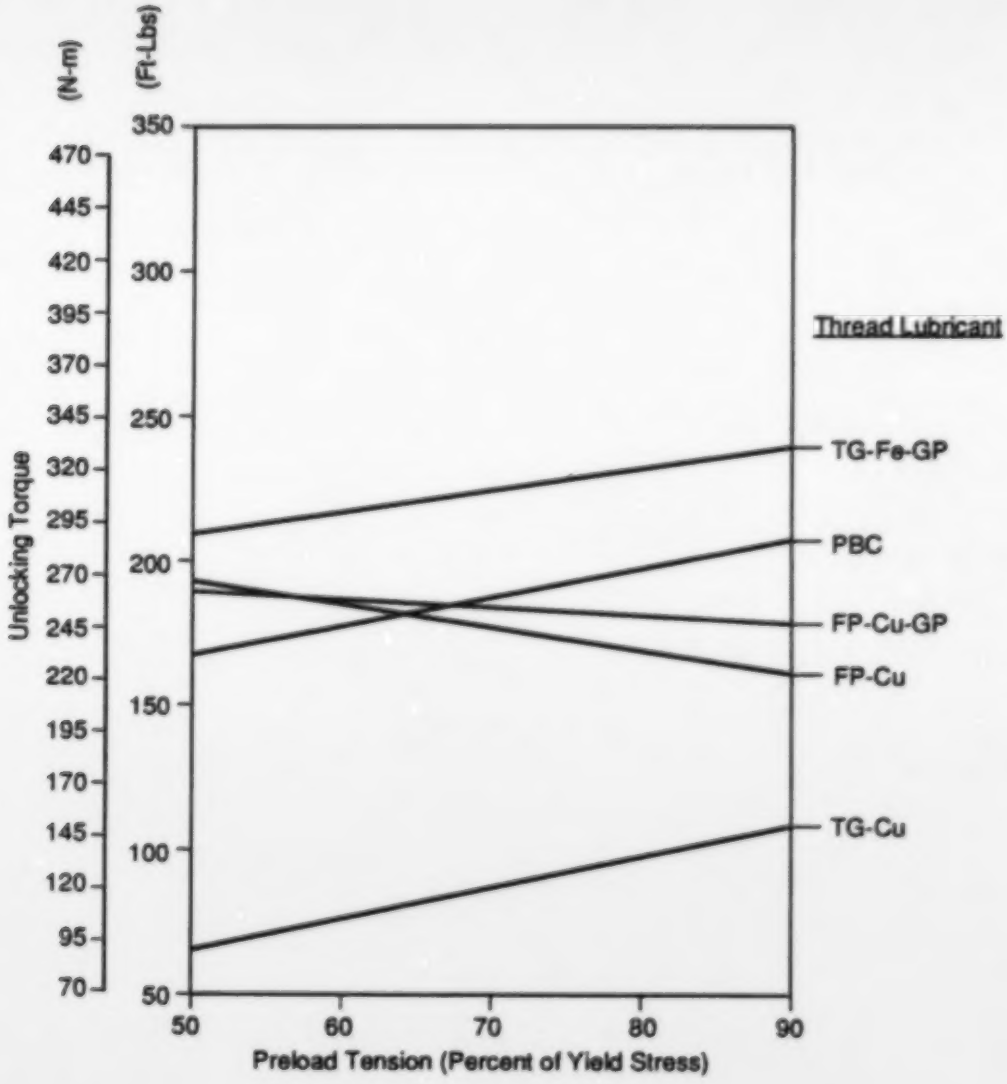


FIGURE 5

UNLOCKING TORQUE VERSUS PRELOAD TENSION - COPPER AND IRON THREAD LUBRICANTS ON ASTM A-193 B7 BOLTING

unlocking torques at 75% YS. The relative rankings obtained are presented in Table VI. As with the preload torque rankings, this data is compared with other rankings in Section 4.1.

Table VI

Lubricant Rating Based on Post-Aging Unlocking Torque

Lubricant	Unlocking Torque (Ft-Lbs @ 75% YS)	Rank (1=best)
TG-Cu	90	1
FP-Cu	170	2
FP-Ni	180	3
CH-Al-GP	180	3
FP-Cu-GP	180	3
PBC	190	4
CH-Ni	198	5
TG-Fe-GP	226	6
TG-Ni	237	7
MOLY	270	8
BLANK	270	8

The unlocking torque data of Appendix 2 were pooled according to metal type and analyzed statistically, as described in Appendix 3. The following were observed from this analysis:

(1) For fasteners initially assembled to 90% YS, the unlocking torques for copper based antiseize compounds are statistically distinguishable from those of other base metals and blank assemblies. That is, the observation that the copper based lubricants have lower torques, is statistically significant, and copper based lubricants offer a torque reduction advantage at the 90% YS aging condition.

(2) For fasteners initially assembled to 90% YS, the unlocking torques for nickel and molybdenum based antiseize compounds and unlubricated assemblies are not statistically distinguishable. That is, no advantage is seen in using nickel or molybdenum based antiseize for torque reduction at the 90% YS aging condition.

(3) For fasteners initially assembled to 50% YS, the unlocking torques for nickel based antiseize compounds are statistically distinguishable from those of other base metals and blank assemblies. That is, the nickel based lubricants offer a torque reduction advantage at the 50% YS aging condition.

(4) For fasteners initially assembled to 50% YS, the unlocking torques for copper based antiseize compounds are statistically distinguishable from those of other base metals and blank assemblies, but to a low degree of confidence (70%). The torque reduction advantage of copper at the 50% YS aging condition is less than that of nickel.

3.2.2 Tensile Strength

The tensile strengths of all bolting specimens were determined after the aging test. Each specimen was held at its ends so that the area of the bolt which was covered by the nuts during the aging test was part of the bolt undergoing extension in the tensile strength test. In every case, the final fracture occurred in or near the middle of the bolt, not in the lubricated areas where SCC effects would be expected to weaken the bolting.

The tensile strength data is presented in Appendix 2. The data were pooled in various ways for statistical analysis, as described in Appendix 3. The following are noted from this analysis:

(1) Pooled yield data for aged bolts is statistically distinguishable from pooled yield data for unaged, unstressed bolts. This is likely the result of stress hardening, resulting in higher yield values for the aged bolts, as all aged bolts were under tension.

(2) Pooled data for aged bolts at 50% YS is not statistically distinguishable from pooled data for aged bolts at 90% YS. That is, the difference in stress hardening between bolts aged at 50% YS and those aged at 90% YS is not significant.

(3) Pooled yield data for aged bolts lubricated with nickel based compounds is marginally statistically distinguishable from yield data for aged, non-lubricated specimens. That is, the nickel lubricated bolting yielded at slightly lower values than the aged blanks. No such difference was seen for the other lubricants.

Because no failures were observed in the lubricated zone, observation (3), above, is not considered significant. It is

unlikely that sufficient lubricant leaked into the fracture area to cause a change in the tensile strength, while the heavily lubricated area did not suffer a similar or greater loss of tensile strength. It is therefore concluded that the lubricants did not cause any significant change in tensile strength relative to unlubricated specimens.

The average increase in tensile strength due to stress hardening is estimated at 4%, based on the data of Appendix 2.

3.3 Inspection for Corrosion Cracking

3.3.1 Visual Inspection

Following disassembly, all bolting was cleaned ultrasonically with mineral spirits as solvent. Three, eight hour cleaning cycles were required to remove residual materials.

After cleaning, the bolting specimens were visually examined for thread damage and lubricant buildup. An estimate of metal to metal contact area was also made, based on the approximate area of bare metal observed in the thread area after removal of the nuts.

A ranking based on area of bare metal contact is presented in Table VII below. As with the torquing data, the Table VII rankings are discussed in more detail in section 4.2.

Significant residual lubricant was found on the thread areas which were under load during the aging test for the copper and iron based lubricants and for the MOLY product. All were thin, hard films except for MOLY, which was a thick, gum-like film. In addition, the iron based product was present as a thick cake in the middle of the bolt. The nickel and aluminum samples showed only a discoloration of the surface.

3.3.2 Microscopic Inspection

The cleaned bolting was microscopically inspected by the Metallurgical Research Department for signs of cracking. No significant cracking was observed for any of the bolting specimens.

The autoclaving conditions, although as severe as those used in other studies, were not severe enough to produce stress corrosion in B7 material. Procedure modifications which would increase the likelihood of observing SCC in controlled tests are discussed in Section 4.3.

Table VII

Lubricant Rating Based on Post-Aging Thread Inspection

Lubricant	Bare Metal Area (% of Thread Area)	Rank (1=best)
TG-Cu	0 - 15	1
CH-Ni	16 - 20	2
FP-Cu	16 - 20	2
TG-Fe-GP	21 - 25	3
MOLY	21 - 25	3
FP-Ni	21 - 25	3
TG-Ni	26 - 30	4
FP-Cu-GP	31 - 35	5
PBC	36 - 40	6
BLANK-Cleaned	> 50	7
Ch-Al-GP	> 50	7
BLANK	>> 50	8

4.0 IMPLICATIONS FOR BRUCE NGS-B OPERATIONS

4.1 Lubricant Selection

As no stress corrosion cracking was observed for any of the test specimens, none of the candidate lubricants could be implicated as accelerators of SCC under the test conditions. This implies that any of the test lubricants could be used without major concern of SCC effects on the short term.

To rate the test lubricants in the absence of SCC data, the rating factors from Tables V, VI and VII were given equal weight and combined to give a cumulative rating according to the formula:

$$\text{Final Rating} = 100 \times ((8-R1) + (8-R2) + (8-R3))/21 \qquad [3]$$

where R1, R2 and R3 are the rating factors from Tables V, VI and VII, respectively.

The Final Rating was chosen to give a rating of 100 if the rank of a lubricant was 1 for all Tables, and to give a rating of 0 if

the rank of a lubricant was 8 on all Tables. The Final Rating data are presented in Table VIII.

Table VIII

Final Rating Factors for Thread Lubricants

Lubricant	Ratings from Tables (By Table Number) V	VI	VII	Final Rating (90 = Best)
TG-Cu	3	1	1	90
FP-Cu	5	2	2	71
MOLY	1	8	3	57
FP-Ni	6	3	3	57
FP-Cu-GP	4	3	5	57
CH-Ni	5	5	2	57
TG-Ni	2	7	4	52
TG-Fe-GP	7	6	3	38
CH-Al-GP	7	3	7	33
PBC	8	4	6	29
BLANK	5	8	8	14

In the absence of a primary rating based on SCC data, the rating data of Table VIII provide a comparison of the thread lubricants based on secondary characteristics such as tightening and unlocking torques and exposed metal in the load zone. The results suggest the following:

(1) Premium grade, copper based thread lubricants are preferred over nickel or molybdenum based lubricants when considering secondary rating factors.

(2) The premium grade nickel products provide about the same rating as the standard molybdenum product, as does one of the general purpose grade copper products.

(3) Other general purpose products and PBC achieve relatively low final ratings.

Because the general purpose products show no major performance advantages relative to premium products, and yet contain trace contaminants of unknown significance, it would be prudent to consider only premium products for bolting lubrication. As

indicated by Table VIII, this means using TG-Cu or FP-Cu where copper based lubricants can be used, or FP-Ni, CH-Ni or TG-Ni if only nickel can be tolerated. (The 5 point difference between TG-Ni and its counterparts is not considered significant.)

If a single supplier is required for both copper and nickel based products, the preferred supplier based on average Table VIII ratings for the premium products is Thread Guard (average = 71), with Fel-Pro a close second (average = 64).

4.2 Bolting Materials

A consideration discussed at the initial meeting on A193-B7 Bolting at BNPD was the use of A193-B7M rather than A193-B7/5/. The B7M material has a slightly higher minimum tempering temperature than B7, and an additional requirement that the heat treatment be done after all machining and forming operations have taken place to guarantee stress relief. The properties of the two materials are compared in Table IX.

Table IX

Mechanical Properties of ASTM A193 B7 and B7M Steels[1]

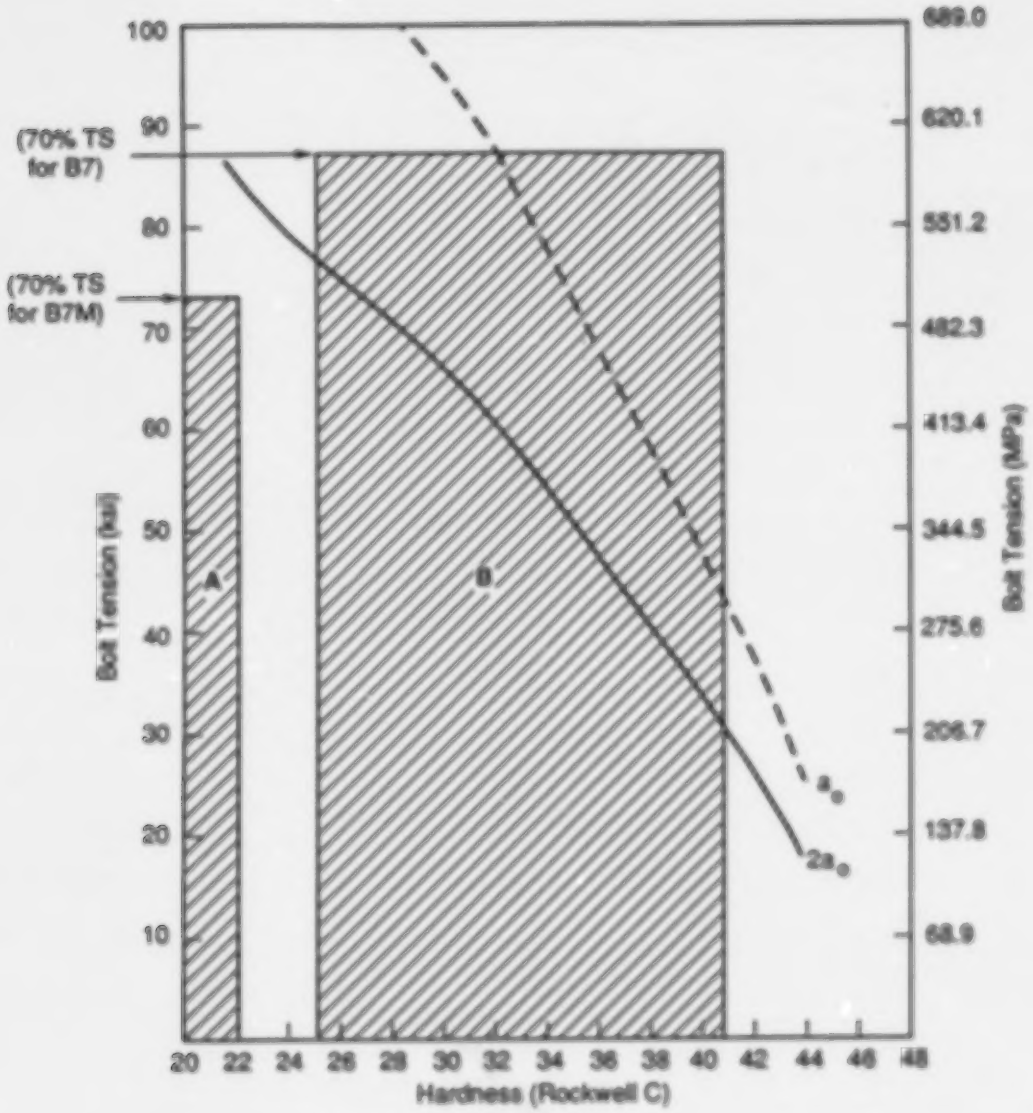
Material	Tensile Strength (MPa)	Yield Strength (MPa)	Minimum Hardness[2] (Vickers)	Maximum Hardness[2] (Vickers)
B7	860	720	263	405
B7M	724[3]	550	221	246

[1] *From ASTM Standard A 1931A 193M - 88.*
[2] *From data supplied by G. Bellamy, Mechanical Research.*
[3] *Note: The 690 Mpa value quoted in Standard A 193 is incorrect. The TS of B7M is therefore 16% less than that of B7, not 20%.*

It is known that the common factors in high strength bolt failures are high bolt hardness and high preload. Therefore, it is desirable to reduce bolt hardness to reduce the likelihood of SCC.

As shown in Figure 6, the hardness range of B7M is lower than that of B7. Further, the B7M range does not intersect theoretical curves representing the minimum tension which would cause SCC, as predicted from fracture mechanics/10/.

As pointed out by N. Baines-Martin (Reference 6), there are formidable design review and possible misuse implications of



Legend

A = Hardness band for A-193 B7M Bolting
B = Hardness band for A-193 B7 Bolting
a_o = Threshold stress for bolt cracking
- crack depth = thread depth
$2a_o$ = Threshold stress for bolt cracking
- crack depth = 2 x thread depth
TS = Tensile strength
70% TS = Typical upper limit

FIGURE 6

THEORETICAL MINIMUM THRESHOLD STRESSES FOR STRESS CORROSION CRACKING, COMPARED WITH HARDNESS BANDS FOR 25 mm DIAMETER ASTM A-193 B7 AND B7M BOLTING (ADAPTED FROM FIGURE 1 OF REFERENCE 10)

using B7M in place of B7 material. However, it is an available alternative material which seems to offer a definite advantage in reducing the likelihood of SCC.

As pointed out by Dr. G. Bellamy, in situations where it is necessary to retain high strength and good resistance to corrosion XM-19 and Inconel 718 could be considered/5/. However, laboratory investigation is required to evaluate these materials.

4.3 Future Corrosion Testing

Because no stress corrosion cracking could be induced in the 1000 hour, 300°C test, the test lubricants could not be rated as SCC accelerators. This is undesirable, given the potential importance of SCC in bolting.

Further study of at least the Fel-Pro and Thred-Guard pairs, plus a MOLY reference material, under more severe conditions, is required to rate the most promising materials relative to SCC acceleration.

Such a study should consider the following changes in the aging technique:

(1) A heating stage after fastener assembly and prior to aging to eliminate lubricant runoff into the autoclave. (The test lubricants contain carrier oils which, when volatilized by heat, can result in a protective, carbonaceous coating on test specimens under autoclaving conditions).

(2) Periodic temperature reduction to room temperature during aging in the autoclave to introduce expansion/contraction stresses, as might be experienced in-plant.

(3) Periodic changes in autoclave water and air to re-establish the initial test environment.

(4) Minimum bolt tension of 90% YS. Specimens at 100% YS and 110% YS should be considered.

(5) Aging to continue until SCC failures are observed.

(6) Increased number of samples per load/lubricant combination.

5.0 CONCLUSIONS

1. Stress corrosion cracking is not induced in normally stressed and highly stressed ASTM B7 bolting specimens by 1000 h exposure to 300°C steam, regardless of the test lubricant applied.

2. Considering only assembly torque, post-test unlocking torque and post-test bare metal contact area in the load zone, premium grade, copper based thread lubricants outperform nickel, aluminum, iron or molybdenum based products.

3. Based on an average rating of premium copper and nickel products, the Thread Guard products marginally outperform those of Fel-Pro. Either Company's products are acceptable.

4. Stress hardening of approximately 4% is induced under the aging test conditions. There is no significant difference between the hardening experienced by bolting tensioned to 50% YS and that of bolting tensioned to 90% YS.

5. For the relatively stiff fastener assemblies of this project, turn angle is a good indicator of bolt elongation and, hence, tension. For such systems, turn angle is more reliable than torque for tension estimates.

6. The carbonaceous coating on as-received bolts acts as a lubricant. However, it aids in assembly only, disassembly is difficult and the metal/metal contact area in the load zone is high.

6.0 RECOMMENDATIONS

1. "Thread Guard Nickel" and "Fel-Pro N-5000 should be considered as the only qualified products for use on threaded fasteners requiring a nickel based thread lubricant.

2. "Thread Guard Copper" and "Fel-Pro N-1000 should be considered as the only qualified products for use on threaded fasteners requiring a copper based thread lubricant.

3. Copper based lubricants should be used in preference to nickel based products where either can be used.

4. Further aging studies are recommended to develop an accelerated aging technique which can discriminate between lubricants on the basis of observed stress corrosion cracking damage.

5. Torque/turn experiments with real or mock-up joints simulating typical fasteners, complete with gasketing and washer materials, are recommended to determine the accuracy, of the turn counting method for bolt preload estimation. Direct elongation measurement techniques should also be tested.

6. Further consideration should be given to the use of ASTM A-193 B7M bolting in place of the B7 product to reduce the potential for SCC.

7.0 ACKNOWLEDGEMENTS

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APPENDIX 1

Yield Stress and Elongation Calculations

A1.1 Yield Stress

Tensile strengths were measured on a Satec Universal Testing Machine (120,000 Lbs capacity) located in the Mechanical Research Department Laboratory. The applicable calibration reference date is August, 1989.

Typical Stress/Strain curves obtained from the Satec machine for as-received bolting are presented on Figure 7 of this Appendix. As noted on the Figure, the Yield Strength average is 18,050 lbs (8,187.3 kgf).

The yield was taken as the point of deviation from the elastic region, as recommended by Dr.G. Bellamy, Metallurgical Research.

The stress area of bolt was measured as 0.1419 in^2 (0.9155 cm^2), based on a diameter calculated as the mean of the root and pitch diameters.

The Yield Stress is therefore: 18,050/0.1419 = 127,202 PSI (877.1 MPa).

A1.2 Elongation

The average Young's modulus, M, for A193 B7 was taken as 30E06 PSI (207 MPa), based on literature values. This is consistent with values obtained from the slopes of the experimental tensile strength curves for as-received specimens. These ranged from 185 MPa to 275 MPa.

The elongation required to tension a bolt to a given percentage yield stress was calculated from the definition of yield stress:

$$\text{Yield Stress} = M \times \text{Ln}(l_f/l_i) \qquad [A1]$$

Where l_f and l_i are final and initial bolt lengths, respectively (see Figure 7 for bolting geometry).

Using l_i = 2.25" (5.72 cm), measured nut-centre to nut-centre on as-received bolts, yields:

$$127{,}202 = 30E06 \times \text{Ln}(l_f/2.25) \text{ at yield point} \qquad [A2]$$

and $$114{,}482 = 30E06 \times \text{Ln}(l_f/2.25) \text{ at 90\% of yield} \qquad [A3]$$

and $$63{,}601 = 30E06 \times \text{Ln}(l_f/2.25) \text{ at 50\% of yield} \qquad [A4]$$

Bolting Material = B - 7 ASTM A193, Nominal Dimensions: 3" x 0.5" Dia
(Avg.) Tensile Area = 0.1419 $(IN)^2$, Approximate Test Length = 2.3", Elongation Rate = 0.001 "/sec.

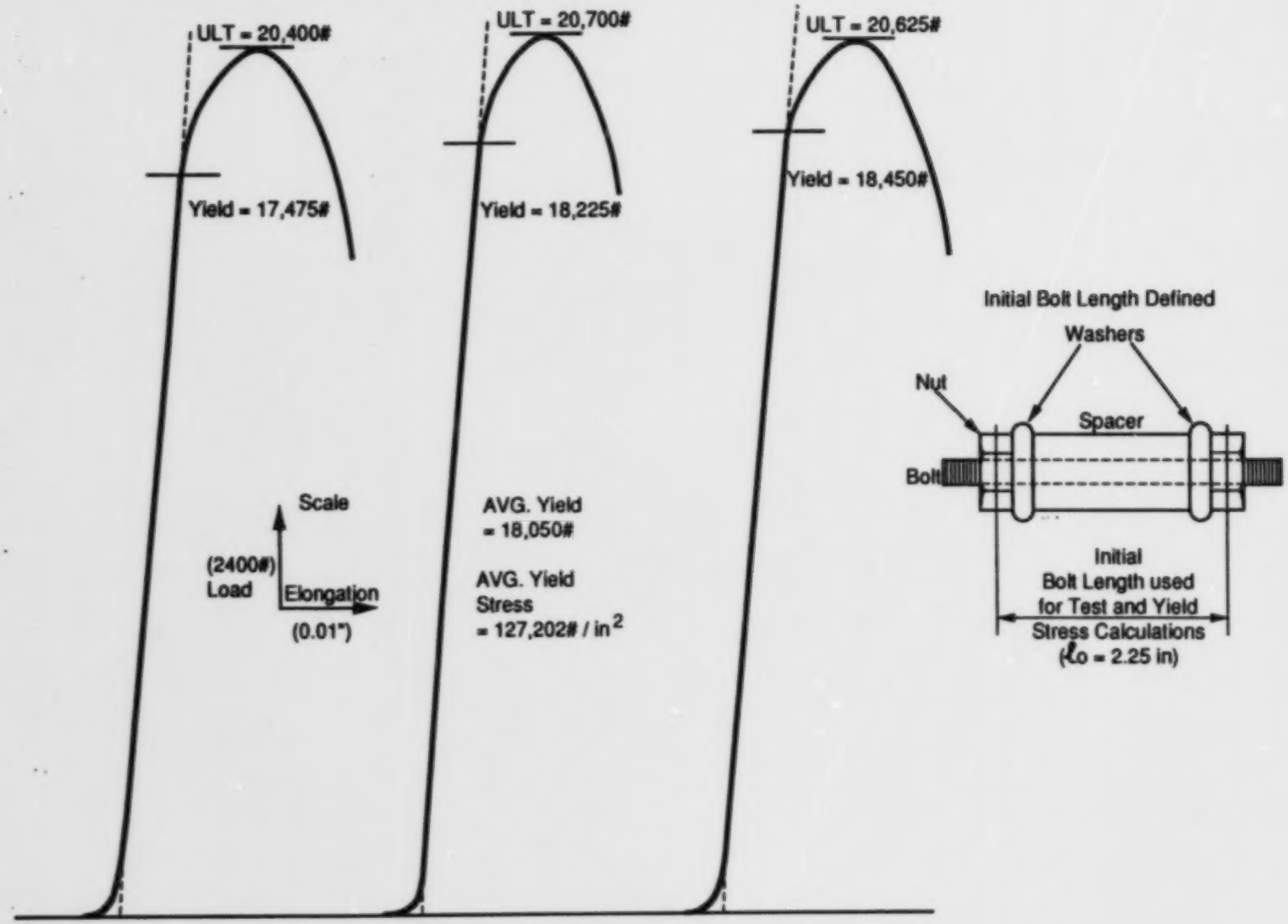

FIGURE 7

TYPICAL SATEC MACHINE TENSILE STRENGTH CURVE OUTPUT FOR AS-RECEIVED B-7 BOLTING

Case A 90% of Yield*

From [A3], l_f = 2.25860 => delta = 0.00860 = 9/1000. (9 thou.)

Case B 50% of yield*

From [A4] l_f = 2.25478 => delta = 0.00478 = 5/1000. (5 thou.)

*Note: Using Engineering Strain in place of Real Strain, and/or determining stress and strain by the 0.2% offset method results in the same elongation estimate of 5 and 9 thou.

From Figure 7, the Tensile Strength and Yield Strength loads are 20,575 Lbs and 18,050 Lbs, respectively. The ratio YS/TS is therefore: 0.88.

(The experimenal data for B7 material is similar to the pusblished data of ASTM Standard A193 for B7, which gives a TS of 125,000 PSI and YS of 105,000 PSI, for a ratio of 0.84).

According to Reference 10, industry practice recommends a tensioning of 60-70% of TS. In terms of YS, the range must therefore be scaled upward by the factor (1/0.88), which yields the tensioning range: 68-80% YS, or about 70-80% of YS.

A1.4 Calculated Friction Coefficients

Average friction coefficients were calculated from the following equation relating applied torque to preload (From Reference 11):

$$T_{in} = F_p(P/2\pi + \mu_r r_t/\mathrm{Cos}(\theta) + \mu_n r_n) \qquad [A5]$$

where T_{in} = applied torque, F_p = preload, P = thread pitch, μ_r and μ_n = nut/bolt and nut/washer friction coefficients, r_t and r_n = thread/nut and nut/washer effective contact radii and θ = the thread half-angle.

It is assumed that $\mu_r = \mu_n$ and θ = 30 degrees.

The preloads were calculated from an accurate estimate of the average yield load (17520 Lbs), based on four tensile strength determinations. The loads used for F_p were therefore 0.9 x 17520 (15768 Lbs) and 0.5 x 17520 (8760 Lbs) for 90% YS and 50% YS cases, respectively.

The following values were obtained by measurement: (1) P = 2 mm = 0.0787 Inch; (2) r_t = root diameter/2 = 10 mm/2 = 5 mm = 0.197 Inch; (3) r_n = inner-most diameter of nut resting on washer/2 = 13.5/2 mm = 6.75 mm = 0.266 Inch.

Experimental torque values were converted to Inch-Lbs from Ft-Lbs by multiplying by 12.

Equation [A5] therefore becomes:

$$12T_{in}/F_p = (0.0125) + (0.197/0.866 + 0.266)\mu \qquad [A6]$$

or:

$$\mu = (12T_{in}/F_p \ -0.0125)/(0.4935) \qquad [A7]$$

or:

$$\mu = 2.026(12T_{in}/F_p \ -0.0125) \qquad [A8]$$

Equation [A8], the F_p values, above, and the preload torque data of Appendix 2 were used to calculate the average friction coefficients presented in Table V of the text.

APPENDIX 2

Torque, Turn and Tensile Strength Data

For torque and tensile data, the data are grouped by major metal type. Sample numbers are interpreted as follows: (1) The first number is the lubricant type, as given in Table 1 of the text, or as indicated below. (2) The letters A and B refer to fasteners tensioned to 90% and 50% of yield stress, respectively. (3) The final number identifies one of two test bolts at a particular tension.

Additional Sample Sets

Sample set 11 = as-received blanks.
Sample set 12 = same as set 11 cleaned with mineral spirits in an ultrasonic bath.
Sample set 13 = same as set 12 lubricated with Fel Pro N-5000
Sample set 14 = same as set 12 lubricated with Fel Pro N-1000

A2.1 Bolting Preloading Torque Data

Major Metal & Sample Number	Preload Torque (Ft-Lbs) Per Bolt	Tension Avg.	Major Metal & Sample Number	Preload Torque (Ft-Lbs) Per Bolt	Tension Avg.
Copper			Nickel		
4A1	85	78	1A1	75	75
4A2	70		1A2	75	
4B1	40	48	1B1	60	50
4B2	55		1B2	40	
5A1	95	85	2A1	95	93
5A2	75		2A2	90	
5B1	50	53	2B1	70	60
5B2	55		2B2	50	
6A1	90	95	3A1	85	88
6A2	100		3A2	90	
6B1	90	90	3B1	40	53
6B2	90		3B2	65	
7A1	75	78	9A1	50	65
7A2	80		9A2	80	
7B1	75	63	9B1	30	33
7B2	50		9B2	35	
8A1	80	80			
8A2	80				
8B1	40	53			
8B2	65				

Major Metal & Sample Number	Preload Torque (Ft-Lbs) Per Bolt	Tension Avg.	Major Metal & Sample Number	Preload Torque (Ft-Lbs) Per Bolt	Tension Avg.
MOS2			Blank		
10A1	50	73	11A1	65	62
10A2	95		11A2	58	
10B1	60	45	11B1	40	45
10B2	30		11B2	50	
			12A1	105	103
13A1	70	70	12A2	100	
13A2	70		12B1	70	70
13B1	60	63	12B2	70	
13B2	65				
			14A1	55	63
			14A2	70	
			14B1	45	45
			14B2	45	

A2.2 Turn Angle to Achieve Preload

Sample	Turn Angle (Degrees)	Sample	Turn Angle (Degrees)
1A1	120	6A1	120
1A2	120	6A2	125
1B1	90	6B1	115
1B2	90	6B2	90
2A1	135	7A1	120
2A2	135	7A2	120
2B1	90	7B1	90
2B2	90	7B2	90
3A1	120	8A1	140
3A2	130	8A2	160
3B1	95	8B1	90
3B2	90	8B2	90
4A1	120	9A1	120
4A2	130	9A2	130
4B1	100	9B1	90
4B2	90	9B2	90
5A1	120	10A1	120
5A2	125	10A2	130
5B1	90	10B1	90
5B2	90	10B2	90

Sample	Turn Angle (Degrees)	Sample	Turn Angle (Degrees)
11A1	135	13A1	120
11A2	130	13A2	120
11B1	90	13B1	90
11B2	90	13B2	85
12A1	140	14A1	120
12A2	130	14A2	120
12B1	90	14B1	90
12B2	90	14B2	90

A2.3 Unlocking Torque Data

Major Metal & Sample Number	Unlocking Torque (Ft-Lbs) Per Bolt	Tension Avg.	Major Metal & Sample Number	Unlocking Torque (Ft-Lbs) Per Bolt	Tension Avg.
Copper			Nickel		
4A1	95	110	1A1	310	335
4A2	125		1A2	360	
4B1	70	68	1B1	105	85
4B2	65		1B2	65	
5A1	110	160	2A1	255	248
5A2	210		2A2	240	
5B1	250	193	2B1	90	70
5B2	135		2B2	50	
6A1	215	208	3A1	160	213
6A2	200		3A2	265	
6B1	210	168	3B1	190	175
6B2	125		3B2	160	
7A1	280	238	9A1	200	210
7A2	195		9A2	220	
7B1	175	210	9B1	110	128
7B2	245		9B2	145	
8A1	235	178			
8A2	120		Blanks		
8B1	240	188			
8B2	135		11A1	320	300
			11A2	280	
MOS2			11B1	240	220
			11B2	200	
10A1	375	295	12A1	170	270
10A2	215		12A2	370	
10B1	130	225			
10B2	320				

A2.4 Tensile Strength Data

Major Metal & Sample Number	Yield Stress (Lbs) Per Bolt	Yield Stress (Lbs) Tension Avg.
As Received		
R1	18000	-
R2	17280	-
R3	17760	-
R4	17040	-
Copper		
4A1	18080	18640
4A2	19200	
4B1	18240	18000
4B2	17760	
5A1	18360	18360
5A2	18360	
5B1	17760	17640
5B2	17520	
6A1	18050	18420
6A2	18790	
6B1	18720	18720
6B2	18720	
7A1	18240	18035
7A2	17830	
7B1	18360	18180
7B2	18000	
8A1	18480	18600
8A2	18720	
8B1	18240	18240
8B2	18240	
MOS2		
10A1	17760	18000
10A2	18240	
10B1	18240	18300
10B2	18360	

Major Metal & Sample Number	Yield Stress (Lbs) Per Bolt	Yield Stress (Lbs) Tension Avg.
Nickel		
1A1	18360	18180
1A2	18000	
1B1	18720	18000
1B2	17280	
2A1	18550	18155
2A2	17760	
2B1	17280	17760
2B2	18240	
3A1	17830	17975
3A2	18120	
3B1	18360	17940
3B2	17520	
9A1	18310	18035
9A2	17760	
9B1	17520	17880
9B2	18240	
Blanks		
11A1	18480	18420
11A2	18360	
11B1	18360	18300
11B2	18240	
12A1	17700	18330
12A2	18960	

APPENDIX 3

Statistical Ananlysis of Torque Data

A3.1 Explanation of Statistical Procedure

The data of Appendix 2 were grouped in various ways as discussed in the text and analyzed statistically. The statistical analyses were carried out by Operations Research (F. Camacho) using the Statistical Analysis System software package (SAS). The SAS program compared data averages to one another using the t-test for significance, as outlined below.

A3.1.1 Theory

In the t-test, the experimental data are used to calculate group means **X1** and **X2**, based on n1 and n2 independent observations, from the populations with calculated standard deviations $\sigma 1$ and $\sigma 2$, respectively.

For similar data it is assumed that $\sigma 1 = \sigma 2 = \sigma$. Under this assumption, the standard error of the difference between the means is given by:

$$\textbf{Standard Error (X1-X2)} = \sigma(1/n1 + 1/n2)^{1/2} \qquad [A3.1]$$

(See, for example, Reference 11, p 61)

The ratio of the difference between the sample means, **(X1-X2)**, and the **Standard Error** defines a quantity known as the t statistic, that is: $t = |(\textbf{X1-X2})/\ \sigma(1/n1 + 1/n2)^{1/2}|$.

Calculated t values are compared to a probability curve known as the t-distribution, which relates to the probability that the two distributions being compared are statistically distinct.

Standard statistical texts, such as Reference 11, provide t tables and details of computational methods.

A3.1.2 Interpretation of Computer Program Output

Most of the computer output on the following Tables is not required to understand the basic results of the statistical analysis. The following data are sufficient:

(1) Symbols CU, MO, NI refer to data sets which are grouped according to base metal composition. N refers to as-received "no-lubricant" blank bolting condition.

(2) The "Parameters" column identifies the statistical comparisons taking place. For example, N vs CU identifies a

comparison between the no-lubricant blank data and that of the copper based lubricants.

(3) The "Pr > T" column represents the probability that the mean values of the data sets being compared are the same. Therefore, 100 x (1 - (Pr > T)) % is the percent probability that the means are different.

3.2 Summary of Probability Data

A3.2.1 Preload Torque

The probability data obtained from the SAS analyses of Pre-Load Torque data (Tables XI and XII) are summarized in Table X.

Table X

Preload Torque Probability Data

Parameter	(Pr> T)	100x(1-(Pr> T)	Interpretation
Table XI data (Tension = 90% YS)			
N vs CU	0.7991	20	no difference
N vs MO	0.5157	48	no difference
N vs NI	0.9331	6	no difference
Table XII data (Tension = 50 YS)			
N vs CU	0.3847	62	no difference
N vs MO	0.4250	58	no difference
N vs NI	0.4516	55	no difference

A3.2.2 Unlocking Torque

The probability data obtained from the SAS analyses of Pre-Load Torque data (Tables XIV and XV) are summarized in Table XIII.

Table XIII

Unlocking Torque Probability Data

Parameter	(Pr > T)	100x(1-(Pr> T)	Interpretation
Table XIV data (Tension 90% YS)			
N vs CU	0.0263	97	signif. difference
N vs MO	0.9394	6	no difference
N vs NI	0.3541	65	possible difference
Table XV data (Tension 50% YS)			
N vs CU	0.2970	70	possible difference
N vs MO	0.9405	6	no difference
N vs NI	0.0584	94	signif. difference

Table XI

Preload Torque Statistics
(Bolt Tension = 90% of Yield)

BOLT_LOA: Analysis of Torque as a function of Lube 3
9:32 Monday, July 30, 1990

General Linear Models Procedure
Class Level Information

Class	Levels	Values
LUBE	4	CU MO N NI

Number of observations in data set = 28

Dependent Variable: TORQUE

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	3	201.0357143	67.0119048	0.32	0.8099
Error	24	5004.9642857	208.5401786		
Corrected Total	27	5206.0000000			

R-Square	C.V.	Root MSE	TORQUE Mean
0.038616	17.82830	14.44092	81.0000000

Source	DF	Type I SS	Mean Square	F Value	Pr > F
LUBE	3	201.0357143	67.0119048	0.32	0.8099

Source	DF	Type III SS	Mean Square	F Value	Pr > F
LUBE	3	201.0357143	67.0119048	0.32	0.8099

Parameter	Estimate	T for H0: Parameter=0	Pr > |T|	Std Error of Estimate
N vs CU	-2.10714286	-0.26	0.7991	8.1872322
N vs MO	8.25000000	0.66	0.5157	12.5062038
N vs NI	0.75000000	0.08	0.9331	8.8432215

Parameter		Estimate	T for H0: Parameter=0	Pr > |T|	Std Error of Estimate
INTERCEPT		80.00000000 B	15.67	0.0001	5.10563633
LUBE	CU	2.85714286 B	0.45	0.6593	6.40025385
	MO	-7.50000000 B	-0.66	0.5175	11.41654990
	N	0.75000000 B	0.08	0.9331	8.84322153
	NI	0.00000000 B	.	.	.

NOTE: The X'X matrix has been found to be singular and a generalized inverse was used to solve the normal equations. Estimates followed by the letter 'B' are biased, and are not unique estimators of the parameters.

Table XII

Preload Torque Statistics
(Bolt Tension = 50% of Yield)

BOLT_LUB: Analysis of Torque as a function of Lube 7
9:32 Monday, July 30, 1990

General Linear Models Procedure
Class Level Information

Class	Levels	Values
LUBE	4	CU MO N NI

Number of observations in data set = 28

Dependent Variable: TORQUE

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	3	1575.000000	525.000000	2.05	0.1338
Error	24	6149.107143	256.212798		
Corrected Total	27	7724.107143			

R-Square	C.V.	Root MSE	TORQUE Mean
0.203907	27.92437	16.00665	57.3214286

Source	DF	Type I SS	Mean Square	F Value	Pr > F
LUBE	3	1575.000000	525.000000	2.05	0.1338

Source	DF	Type III SS	Mean Square	F Value	Pr > F
LUBE	3	1575.000000	525.000000	2.05	0.1338

Parameter	Estimate	T for H0: Parameter=0	Pr > |T|	Std Error of Estimate
N vs CU	-8.03571429	-0.89	0.3847	9.0749167
N vs MO	11.25000000	0.81	0.4258	13.8621643
N vs NI	7.50000000	0.77	0.4516	9.8020304

Parameter		Estimate	T for H0: Parameter=0	Pr > |T|	Std Error of Estimate
INTERCEPT		48.75000000 B	8.61	0.0001	5.65920486
LUBE	CU	15.53571429 B	2.19	0.0385	7.09418874
	MO	-3.75000000 B	-0.30	0.7695	12.65436678
	N	7.50000000 B	0.77	0.4516	9.80203036
	NI	0.00000000 B	.	.	.

NOTE: The X'X matrix has been found to be singular and a generalized inverse was used to solve the normal equations. Estimates followed by the letter 'B' are biased, and are not unique estimators of the parameters.

Table XIV

Unlocking Torque Statistics
(Bolt Tension = 90% of Yield)

BOLT_UA: Analysis of Torque as a function of Lube 1
9:32 Monday, July 30, 1990

General Linear Models Procedure
Class Level Information

Class	Levels	Values
LUBE	4	CU MO N NI

Number of observations in data set = 22

Dependent Variable: TORQUE

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	3	48129.31818	16043.10606	3.82	0.0281
Error	18	75640.00000	4202.22222		
Corrected Total	21	123769.31818			

R-Square	C.V.	Root MSE	TORQUE Mean
0.388843	28.60843	64.82455	226.590909

Source	DF	Type I SS	Mean Square	F Value	Pr > F
LUBE	3	48129.31818	16043.10606	3.82	0.0281

Source	DF	Type III SS	Mean Square	F Value	Pr > F
LUBE	3	48129.31818	16043.10606	3.82	0.0281

Parameter	Estimate	T for H0: Parameter=0	Pr > |T|	Std Error of Estimate
N vs CU	121.50000000	2.42	0.0263	50.21298802
N vs MO	5.00000000	0.08	0.9394	64.82455495
N vs NI	48.75000000	0.95	0.3541	51.24830662

Parameter		Estimate	T for H0: Parameter=0	Pr > |T|	Std Error of Estimate
INTERCEPT		251.2500000 B	10.96	0.0001	22.91893928
LUBE	CU	-72.7500000 B	-2.37	0.0294	30.74898372
	MO	43.7500000 B	0.85	0.4045	51.24830621
	N	48.7500000 B	0.95	0.3541	51.24830621
	NI	0.0000000 B	.	.	.

NOTE: The X'X matrix has been found to be singular and a generalized inverse was used to solve the normal equations. Estimates followed by the letter 'B' are biased, and are not unique estimators of the parameters.

Table XV

Unlocking Torque Statistics
(Bolt Tension = 50% of Yield)

BOLT_UB: Analysis of Torque as a function of Lube 5
9:32 Monday, July 30, 1990

General Linear Models Procedure
Class Level Information

Class	Levels	Values
LUBE	4	CU MO N NI

Number of observations in data set = 22

Dependent Variable: TORQUE

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	3	32361.07955	10787.02652	2.47	0.0951
Error	18	78671.87500	4370.65972		
Corrected Total	21	111032.95455			

R-Square	C.V.	Root MSE	TORQUE Mean
0.291455	42.09671	66.11097	157.045455

Source	DF	Type I SS	Mean Square	F Value	Pr > F
LUBE	3	32361.07955	10787.02652	2.47	0.0951

Source	DF	Type III SS	Mean Square	F Value	Pr > F
LUBE	3	32361.07955	10787.02652	2.47	0.0951

Parameter	Estimate	T for H0: Parameter=0	Pr > |T|	Std Error of Estimate
N vs CU	55.00000000	1.07	0.2970	51.2093335
N vs MO	-5.00000000	-0.08	0.9405	66.1109652
N vs NI	105.62500000	2.02	0.0584	52.2653071

Parameter		Estimate	T for H0: Parameter=0	Pr > |T|	Std Error of Estimate
INTERCEPT		114.3750000 B	4.89	0.0001	23.37375591
LUBE	CU	50.6250000 B	1.61	0.1238	31.35918426
	MO	110.6250000 B	2.12	0.0485	52.26530710
	N	105.6250000 B	2.02	0.0584	52.26530710
	NI	0.0000000 B	.	.	.

NOTE: The X'X matrix has been found to be singular and a generalized inverse was used to solve the normal equations. Estimates followed by the letter 'B' are biased, and are not unique estimators of the parameters.

A3.2.3 Yield Stress

The probability data obtained from the SAS analyses of yield stress data (Tables XVII and XVIII) are summarized in Table XVI.

Table XVI

Yield Stress Probability Data

Parameter	(Pr> T)	100x(1-(Pr> T)	Interpretation
Table XVII data (Pooled Tension data)			
N vs CU	0.7294	27	no difference
N vs MO	0.4633	54	no difference
N vs NI	0.1072	89	possible
Table XVIII data (aged, 90% YS Tension and 50% YS Tension vs unaged, untensioned bolting)			
None vs A	0.0026	99.7	signif. difference
None vs B	0.0123	98.8	signif. difference

Table XVII

Tensile Strength Statistics
(Blank vs Lubricated Bolting)

SAS 10:22 Thursday, July 26, 1990

General Linear Models Procedure
Class Level Information

Class	Levels	Values
LUBE	4	CU MO N NI

Number of observations in data set = 44

Dependent Variable: STRESS

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	3	918305.7955	306101.9318	1.90	0.1445
Error	40	6432148.7500	160803.7188		
Corrected Total	43	7350454.5455			

R-Square	C.V.	Root MSE	STRESS Mean
0.124932	2.206732	401.0034	18171.8182

Source	DF	Type I SS	Mean Square	F Value	Pr > F
LUBE	3	918305.7955	306101.9318	1.90	0.1445

Source	DF	Type III SS	Mean Square	F Value	Pr > F
LUBE	3	918305.7955	306101.9318	1.90	0.1445

Parameter		Estimate	T for H0: Parameter=0	Pr > |T|	Std Error of Estimate
INTERCEPT		17990.62500 B	179.46	0.0001	100.2508475
LUBE	CU	292.87500 B	2.18	0.0354	134.5006259
	MO	159.37500 B	0.71	0.4812	224.1677098
	N	369.37500 B	1.65	0.1072	224.1677098
	NI	0.00000 B	.	.	.

NOTE: The X'X matrix has been found to be singular and a generalized inverse was used to solve the normal equations. Estimates followed by the letter 'B' are biased, and are not unique estimators of the parameters.

Dependent Variable: STRESS

Parameter	Estimate	T for H0: Parameter=0	Pr > |T|	Std Error of Estimate
N vs CU	76.50000000	0.33	0.7294	219.638602
N vs MO	210.00000000	0.74	0.4633	283.552216
N vs NI	369.37500000	1.65	0.1072	224.167710

Table XVIII

Tensile Strength Statistics
(Non-Preloaded Bolting vs Preloaded and Aged Bolting)

SAS 14:38 Thursday, July 5, 1990 2

General Linear Models Procedure

Dependent Variable: STRESS

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	5	2790660.341	558132.068	3.50	0.0098
Error	42	6693639.659	159372.373		
Corrected Total	47	9484300.000			

R-Square	C.V.	Root MSE	STRESS Mean
0.294240	2.203476	399.2147	18117.5000

Source	DF	Type I SS	Mean Square	F Value	Pr > F
LUBE	3	1064951.250	354983.750	2.23	0.0990
LOAD	2	1725709.091	862854.545	5.41	0.0081

Source	DF	Type III SS	Mean Square	F Value	Pr > F
LUBE	3	918305.795	306101.932	1.92	0.1409
LOAD	2	1725709.091	862854.545	5.41	0.0081

Parameter		Estimate	T for H0: Parameter=0	Pr > |T|	Std Error of Estimate
INTERCEPT		17150.62500 B	57.28	0.0001	299.4110214
LUBE	CU	292.87500 B	2.19	0.0343	133.9006794
	MO	159.37500 B	0.71	0.4791	223.1677990
	N	369.37500 B	1.66	0.1053	223.1677990
LOAD None vs	A	924.54545 B	3.20	0.0026	288.6317477
	B	755.45455 B	2.62	0.0123	288.6317477

NOTE: The X'X matrix has been found to be singular and a generalized inverse was used to solve the normal equations. Estimates followed by the letter 'B' are biased, and are not unique estimators of the parameters.